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A METHOD FOR MEASURING THE DENSITY OF MOLTEN GLASS

BY

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DEGREE OF BACHELOR OF SCIENCE

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A METHOD FOR MEASURING THE DENSITY OF MOLTEN GLASS.

I. INTRODUCTION

All density determinations for glass have been made at temperatures below the softening point. It was the purpose of the present investigation to determine the density of molten glass at temperatures up to 1400° C.

The literature on the determination of densities of molten liquids at high temperatures revealed only two methods of importance. Both were applications of the principle of Archimedes. Day, Sosman and Hostetter,* in their "Determination of Mineral and Rock Densities at High Temperatures", developed a very expensive and elaborate apparatus which was only fairly satisfactory. The other work was by F. M. Jaeger,** who determined densities from the loss in weight of a platinum bob (immersed in the melt) due to the buoyant action of the fluid glass. The practical application of this method is not as simple as it at first appears, and the corrections for surface tension and viscosity, etc., necessary for the calculation of density would require their determinations.

*Amer. Jour. of Sci. ser.4, v.37, p. 1-38.
**See Über die Temperaturabhängegkeit der molekularen freien Oberflächenergie von Flüssigkeiten im Temperaturbereich von -80 bis -1650°C., by Dr. F. M. Jaeger.

II. THE METHOD EMPLOYED

After due consideration of the experimental difficulties and opportunities for error in the application of the principle of Archimedes, it was decided to use the pycnometer method, i.e., the determination of the weight of a known volume of liquid glass.

For this method of determination the procedure is as follows: a pycnometer of known volume and weight is evacuated to a pressure of 0.2 to 0.3 mm. of mercury, and sealed off in such a manner that the vacuum is not destroyed until the pycnometer is totally immersed in the liquid glass. The temperature of the pycnometer when immersed is accurately determined by some suitable means. When filled, the pycnometer is removed and the weight of the glass therein determined. From the calculated volume of the pycnometer at the filling temperature and the weight of the glass therein, the density is obtained by the equation, density = $\frac{\text{mass}}{\text{volume}}$.

The pycnometer must be constructed of a material which has the following properties: (1) melting point above 1400°C.; (2) strength and rigidity at 1400° sufficient to retain shape; (3) known coefficient of expansion up to 1400°C.; (4) ability to resist corrosive action of melt without appreciable change in volume; (5) a non-wetting surface to the melt, or such a different expansion as to make cleaning the pycnometer easy and complete before the final weighing. The cost and machining properties are also important considerations.

The properties of nickel make it a desirable material for this purpose. There has been, however, no accurate determination made of its linear expansion above 1000°C., but it was decided to

use the values given in Landolt, Bornstein, and Roth, as determined by C. Holborn and Day* between 300 and 1000° C., and by extrapolation calculate the expansion at the higher temperatures.

Since the coefficient of expansion of nickel is larger than that of glass, the pycnometer on cooling will either rupture or, if the glass is sufficiently fluid, the pressure will eject a small quantity of it from the opening. If liquid glass is ejected provision must be made to collect it. The opening thru which glass enters the cylinder must be of such shape that it can be quickly cleaned off when taken from the melt. With this point in mind, the first pycnometer was designed as shown in figure I, Plate I. The hole in the riser tube, at the top, thru which the glass enters the cylinder is 2 mm. in diameter. This size was determined by calculation from viscosity values determined in this laboratory for a lead glass at 1350°C. This diameter is sufficient to allow the pycnometer which has a volume of about 70cc., to fill under atmospheric pressure in 30 minutes. A copper tube, 3/16 inches in diameter and 20 inches long, was braised on to this riser tube for a double purpose. First, as a means by which the pycnometer could be sealed off when evacuated, and second, to afford a means of breaking the vacuum seal underneath the surface of the glass melt. If the pycnometer is pushed quickly under the surface, the seal remains intact until the copper tube melts. The pressure of the atmosphere will then force the glass into the pycnometer.

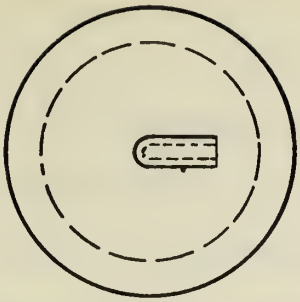
The pycnometer was evacuated by connecting the copper tube to a vacuum line by means of a short length of rubber tubing. When the evacuation was completed, a pinch-cock was clamped on the rubber tubing. In order to see if the pycnometer was holding the vacuum,

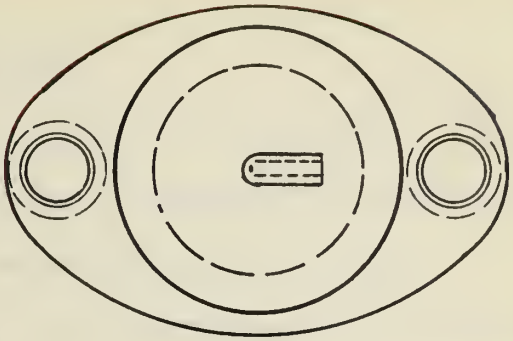
*Ann.d.Phys. v.4 (4) p. 104 (1901)

the pinch-cock was unclamped, and the action of the manometer in the vacuum line observed. Some difficulty was encountered in obtaining a pressure of 0.3mm. but by filling the space between the top and cylinder with solder, the pycnometer was made to hold this pressure indefinitely. This use of solder for obtaining a vacuum tight joint is not to be recommended however, for on melting, some of the solder would doubtlessly flow into the pycnometer and change its volume.

Volume determinations of the pycnometer were made by dividing the difference in weight of the pycnometer when empty and the weight when filled with mercury, by the density of mercury at the room temperature.

PLATE I.





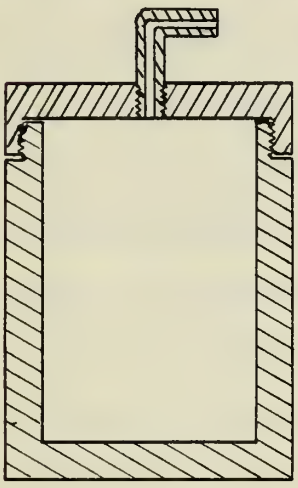
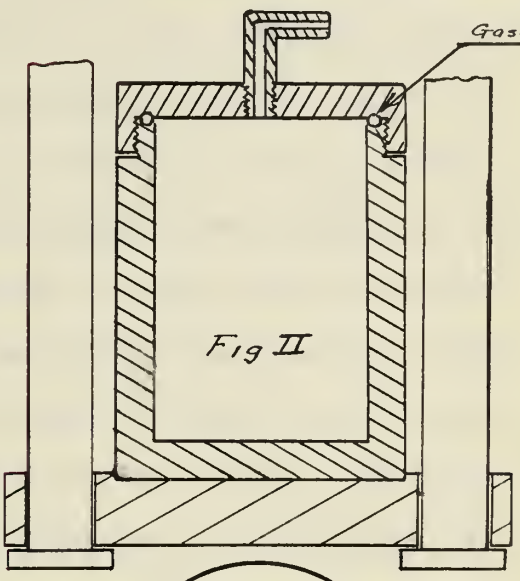


Fig I



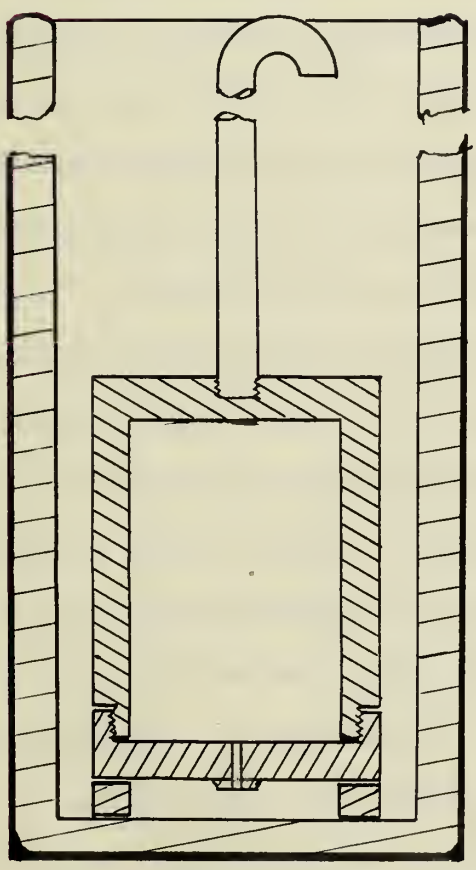


Fig III

Scale 3/4" to 1"

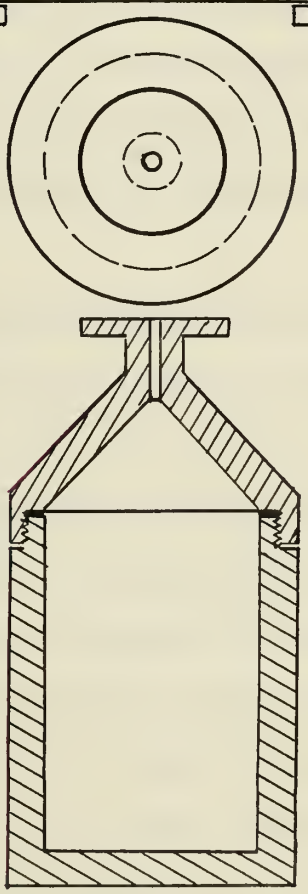


Fig IV

III. FIRST EXPERIMENT

In the first experiment the melt was a lead glass. The glass pot was about 10 inches deep and had a capacity of a little over 3 liters. Temperature readings were taken with a Fery Radiation pyrometer, which was focused on the surface of the melt, so that the temperature observed was that of the coldest part of the melt. The temperature was first raised to 1400°C. and held at that point for one hour to free the melt from bubbles. Then at a surface temperature of 1380°, the pycnometer was quickly lowered into the melt. The vacuum seal appeared to hold, and the copper tube came off as was expected. The temperature was held at 1380° for half an hour. An effort was then made to lift the pycnometer from the pot with a pair of steel tongs but the tongs softened at this temperature and a firm grip on the pycnometer could not be obtained. In order to remove the pycnometer, it was necessary to take the pot out of the furnace and pour out the molten glass. The pycnometer came out in four pieces. The nickel had fractured when it was plunged into the glass.

From this first run, although unsuccessful, several important facts were learned. First, the nickel must be heated before plunging into the molten glass. Second, the surface temperature of the glass in such a pot lagged behind the temperature in the bottom by about 15C°C. A close examination of the pycnometer fragments revealed evidences of melting. Third, a cradle or support by which the pycnometer could be lowered into and taken from the melt was necessary. Furthermore, the use of solder to make the pycnometer vacuum tight is undesirable, because the solder vaporizes at these high temperatures filling the glass and pycnometer with its gases.

IV. SECOND EXPERIMENT

The equipment in this experiment was substantially the same as that in the first. The only changes were the cradle, in which the pycnometer rested while in the melt, and the addition of a copper gasket at the point A in Fig. II, Plate I. This gasket eliminated the necessity for the use of solder. The Féry Radiation pyrometer was again used for temperature determinations, but the surface temperature in this experiment was held at 1250° C. It was estimated that the temperature 2 inches below the surface, the level to which the riser tube came, was at least 50° higher. The pycnometer was heated to a cherry red, and at that temperature was lowered into the melt. After 30 minutes, during which time the temperature was held constant, the cradle and pycnometer were lifted from the pot. Examination of the pycnometer revealed that it had not filled with glass. No doubt the vacuum had been destroyed when the pycnometer was heated before plunging into the melt. As all possible care had been taken with this operation, it was decided that the arrangement used to seal off the pycnometer was not practical, and that some radical change in design and method of procedure were necessary if results were to be obtained with this method.

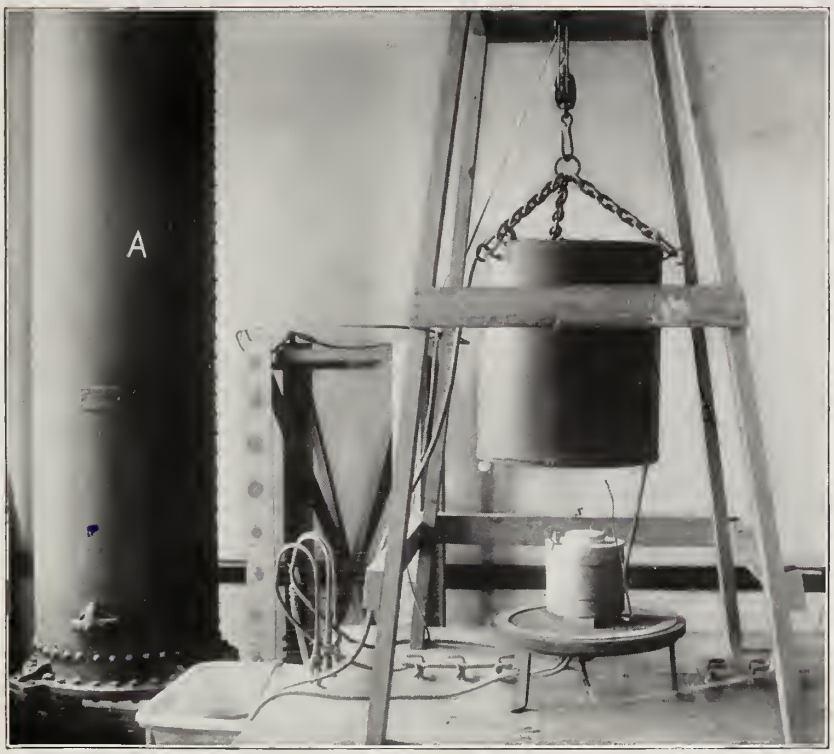
The preheating of the nickel, however, did eliminate the danger of the pycnometer breaking when it was lowered into the melt. Nickel proved itself a desirable material for pycnometer construction as it cleaned easily and thoroughly.

V. THIRD EXPERIMENT

In view of the difficulties encountered in the first two experiments, it was decided that a change in method was necessary. For this reason a new method was devised which was as follows: a small electrically heated pot furnace containing the pycnometer and glass was put under a large metal bell jar capable of being evacuated to a pressure of less than 1.0 mm. of mercury. (See Plates II and III.) The temperature of the glass pot was then gradually raised, and at the same time the pressure in the bell jar gradually reduced. When the molten glass had become practically free from occluded gases, and had reached the desired temperature, the vacuum was broken and the glass was forced into the pycnometer under a pressure of one atmosphere. The pycnometer was then removed and weighed as outlined in the preceding experiments. Fig. II shows the arrangement thru which the pot and pycnometer could be observed while under the bell jar. This was necessary in order that the progress of the fining operation could be observed. Results obtained by this method would give the density of practically gas-free glass.

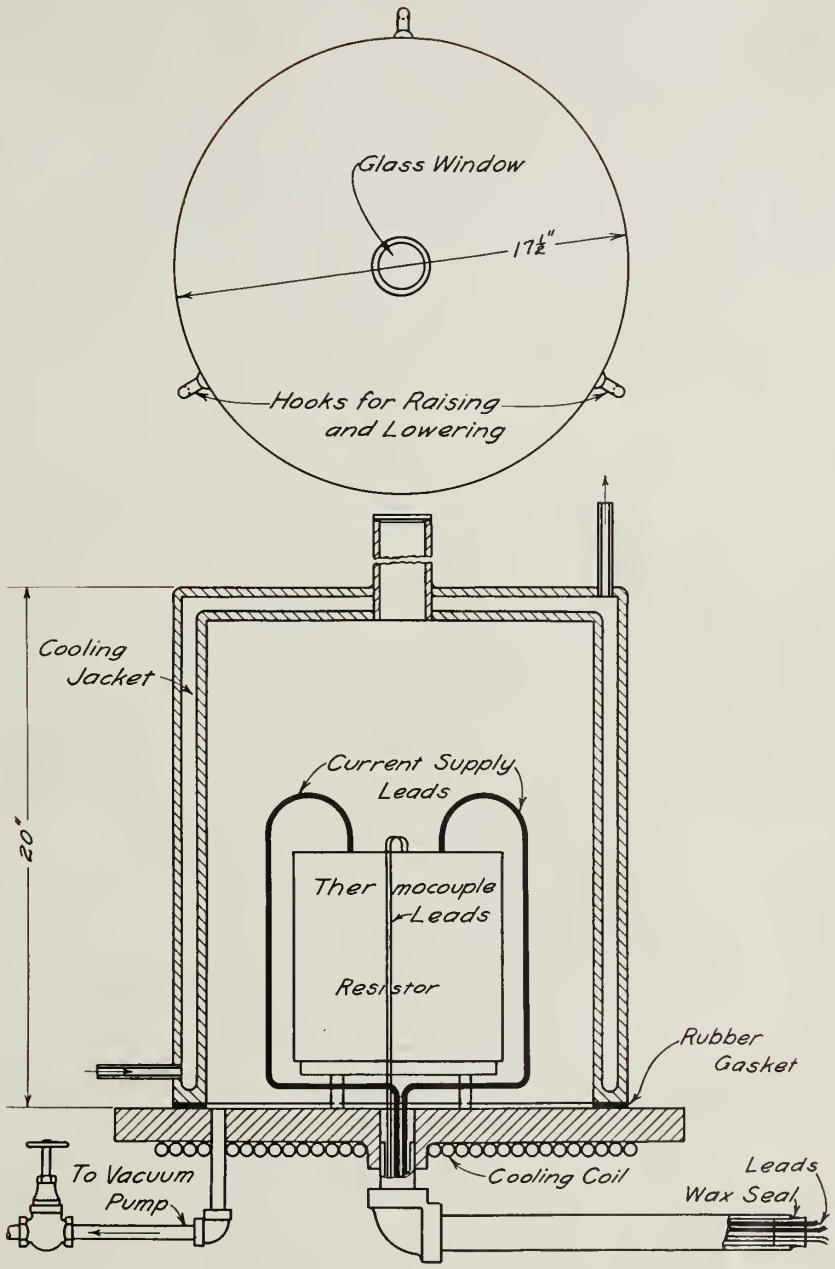
This new method necessitated a change in the design of the pycnometer. The riser tube was eliminated and the top made of one solid piece. The opening was enlarged to 4mm. in diameter. Provision for lifting the cylinder from the pot, was made by means of a nickel rod, hooked at one end and threaded at the other end which screwed into the base of the pycnometer. (See Fig. III, Plate I.)

PLATE II.



Note. The tank A is not a part of the equipment. The coil to the right of the tank is the heating coil rheostat.

PLATE III.


Glass Window
17½"
Hooks for Raising and Lowering
Cooling Jacket
20"
Current Supply Leads
Thermocouple Leads
Resistor
Rubber Gasket
To Vacuum Pump
Cooling Coil
Leads
Wax Seal

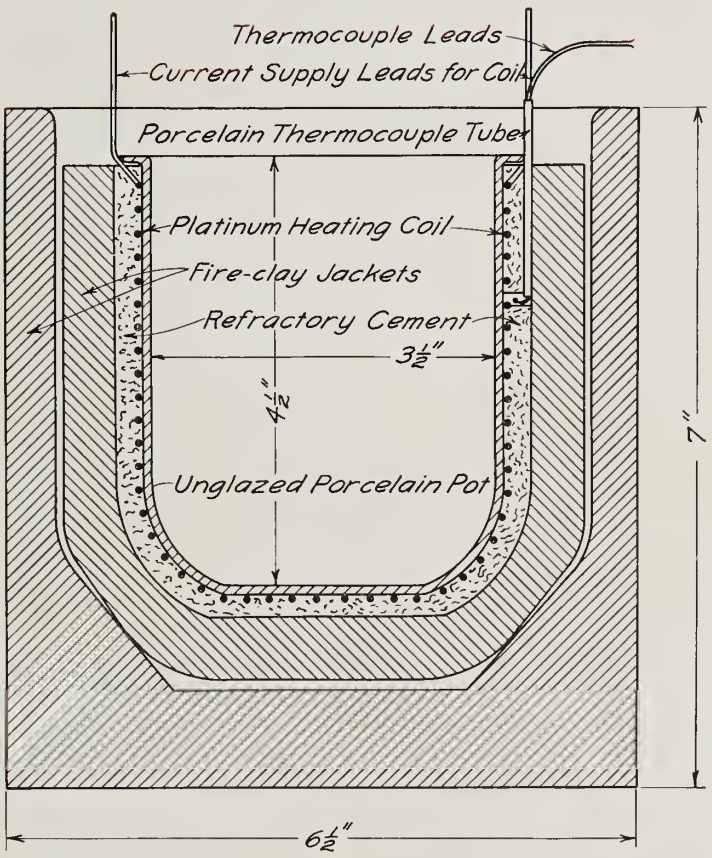
The pot was heated by passing a current thru a platinum coil which was wound on the outside of the alundum cylinder in which the pot rested. Temperature was determined by means of a thermo-couple of platinum and platinum-rhodium alloy, the hot junction of which was between the outer wall of the pot and the inner wall of the alundum cylinder, midway between the top and bottom of the pot. (Plate IV. is cross-section view of the furnace used if the pot dimensions are changed to 3 1/4" wide and 7 1/2" high. The platinum heating coil should be shown as circling the alundum or refractory cement.) It was checked by means of a Leeds-Northrup optical pyrometer, which was sighted on the surface of the melt.

The pot and pycnometer are shown in Fig. III, Plate I. It will be observed from the figure that the pycnometer opening is near the bottom of the pot. The pycnometer rested upon two nickel pellets, which raised the opening at least a quarter-inch from the bottom of the pot. When the pycnometer was lifted from the pot in this inverted position, atmospheric pressure on the opening held the glass in the cylinder.

One run with this arrangement revealed its most serious defect. No matter how carefully the temperature and pressure were controlled, gases coming out of the glass and the nickel were certain to be trapped in the upper part of the pycnometer. This resulted in a partially filled cylinder.

The method of lifting the cylinder from the pot, and all other arrangements were satisfactory, with the exception of heat distribution and temperature control. The surface temperature of the glass as determined by the optical pyrometer lagged 150 degrees behind the temperature at the thermo-couple junction. No doubt the temperature in the bottom of the pot was also considerably higher than that at the thermo-couple.

PLATE IV.


Thermocouple Leads
Current Supply Leads for Coil
Porcelain Thermocouple Tube
Platinum Heating Coil
Fire-clay Jackets
Refractory Cement
3½"
4½"
Unglazed Porcelain Pot
7"
6½"

VI. FOURTH EXPERIMENT

The pycnometer shown in Fig. IV, Plate I, was made especially for this experiment. The sloping top facilitates the escape of any gases liberated in the pycnometer. The flat platform serves a double purpose, in that it affords a means of collecting all glass ejected on cooling, and can be gripped by a pair of tongs.

A lead glass was also used in this run. It was fined by alternately increasing the temperature and diminishing the pressure. Fining was considered complete when no more gas bubbles could be seen in the glass. The pressure had been reduced to 0.3mm. and the temperature increased to 1350°C. when the fining was considered complete. During the fining process, gas bubbles could be seen arising from the opening in the pycnometer, which showed that the sloping cap was functioning as expected. Care must be taken not to reduce the pressure too quickly or the occluded gases will cause the glass to froth and run over the sides of the pot. When fining was complete the vacuum was broken. The glass then sank to a new level. In a few minutes the level was still lower, and a glance at the ammeter in the heating coil circuit showed a short circuit in that line. The bottom of the pot had failed, due to the corrosive action of the lead glass and the weight of the pycnometer. Furthermore the temperature at the bottom must have been considerably higher than that at the thermo-couple, for the nickel on the bottom of the pycnometer had melted. It is also possible that at this high temperature lead from the glass formed a low temperature alloy with the nickel. The entire cylinder showed evidences of corrosion, and had the appearance of having been exposed to the action of lead vapor.

This experiment showed the necessity of some arrangement whereby the temperature of all parts of the pot could be measured.

As no more time was available the work was discontinued at this point.

VII. SUMMARY AND CONCLUSIONS

Although unsuccessful in accomplishing the main purpose of the investigation, a few facts have been ascertained which are worthy of notice:

(1) A nickel pycnometer is not suitable for work with lead glasses, but would probably be satisfactory for other glasses.

(2) The design of the pycnometer used in the last experiment is satisfactory and is recommended.

(3) The actual temperature of the glass in the pycnometer should be measured.

In conclusion I wish to thank Professor E. W. Washburn for suggesting this work and for his kind interest and suggestive criticism during its progress. I am also indebted to Dr. E. N. Bunting for his help and guidance in the laboratory.

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